

HYDRATION RATE OF HOPEWELL

OBSIDIAN IN OHIO

SENIOR THESIS

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the degree of Bachelor of Science by

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## ABSTRACT

Evidence indicates that obsidian hydration rate dating can be used to date the time of exposure of a fresh surface of obsidian. An estimated site temperature constant for Ohio and constants derived by artificially hydrating obsidian from the same source as the obsidian found in Ohio were used to determine that obsidian from a Hopewell mound in Ohio was chipped  $227 \pm 62$  AD. This date corresponds to radiocarbon dates of  $230 \pm 80$  AD and  $280 \pm 55$  AD from a Hopewell occupation site near the mound.

## INTRODUCTION

Obsidian is a homogenous natural glass with a propensity to hydrate when surfaces are exposed to the atmosphere. When a thin section is cut normal to the surface of hydration and viewed with crossed nicols under a polarizing microscope, a birefringent band can be observed. This zone of birefringence corresponds to the depth of penetration of water by the glass; by measuring the distance into the obsidian that water has diffused, one can conclude the time it has taken for the hydration rim to form. This technique was first introduced by Friedman and Smith (1960). Hydration rates were calibrated against radiocarbon dates of worked obsidian artifacts from various archaeological sites. The hydration rate followed the formula:

$$D = (kt)^{1/2}$$

where D = thickness of rim in microns, t = time in years, and k = a constant (based on radiocarbon dates). As various researchers attempted the technique with material from their archaeological sites, it soon became apparent that this expression did not satisfactorily describe the hydration phenomena (Johnson, 1969; Meighan et al., 1968; Katsui and Kondo, 1965; Clark, 1961). Questions were raised and it was soon realized that this process

of water uptake by volcanic glass was not as straightforward as anticipated.

It became evident that the rate of obsidian hydration could depend on several factors; the chemical composition of the glass; the temperature at which obsidian was hydrated; the chemical environment of hydration; and the water vapor pressure or relative humidity. These problems have been examined and evaluated, and although further research needs to be conducted, obsidian hydration rim dating has become a valuable, inexpensive tool for the modern archaeologist.

The purpose of this study is to measure the amount of hydration in obsidian samples found in a Hopewell site in Ohio, and to compare the results with known radiocarbon dates from that site.

#### BACKGROUND ON THE HOPEWELL CULTURE

Obsidian has long been used by native populations to make tools and sharp weaponry for either utilitarian use or as ceremonial objects. In some instances elaborate trade exchanges were constructed to obtain the precious glass and other materials. Obsidian is an ideal material to use when one is trying to reconstruct ancient trade patterns because it is durable, source specific (Griffin et al., 1969), and able to be dated. Its

appeal to the Indian populations stemmed from its workability and beauty. Obsidian possesses a perfect conchoidal fracture and strength which makes it easy to fashion by skilled artisans using percussion flaking. Also inherent in obsidian is a natural beauty manifested by its availability in an array of hues of black, brown, green and red, some containing various internal structures and banding. The early Indians highly prized so useful and attractive a material.

Obsidian is locally abundant in the western United States, but does not occur in the eastern regions. Most obsidian found in Ohio is discovered in remains of ceremonial structures built by the Hopewell people, who enjoyed a high level of culture in the Scioto River area from approximately 300 BC to 600 AD. The Hopewell had an extensive trade network that involved not only obsidian from 1500 miles west of their establishments, but also copper from the Great Lakes, shells from the Atlantic and Gulf coasts, mica from the Appalachians, as well as other imports from various distant localities. It can be seen from their reverent treatment of these exotic imports that the trade network system played an important part in their social, economic and religious lives. At the source areas, control of quarries by a tribe could mean wealth, political strength, or constant conflict for dominance. It is



also possible that some quarries were considered neutral ground protected by religious beliefs, as perhaps was the Flint Ridge area in Ohio, an important flint quarry.

Outcrops of volcanic glass are accessible and easily quarried. The pieces were fashioned into small cylindrical fragments on the spot for easy transport and later reworked into whatever shape was desired. Evidence of this subsequent working at the site of burial is seen by the amount of chippage and debris found in the remains. The Hopewell people lived in small semi-permanent towns, and constructed mounds and earthworks as ceremonial centers to serve large areas. In the case of the Hopewell culture, the chief aim of the trade network for obsidian appears to be exclusively for the production of ceremonial objects for the dead. Since the obsidian artifacts and flakes were found within the mounds, rapid burial after their manufacture is indicated. One of the notable features of the decline of the Hopewell culture was the breakdown of the exchange system (Prufer et al., 1965).

#### THE NATURE OF OBSIDIAN AND HYDRATION MECHANISMS

There are many types of obsidian: rhyolitic, latitic, dacitic, andesitic, trachytic, as well as others. Most are rare with the exception of rhyolitic, which is widespread, and

therefore was the type most commonly used by ancient cultures. Obsidian is amorphous, isotropic and shows no double refraction between crossed nicols unless it is devitrified or under strain. Properties such as specific gravity and refractive index vary according to composition; average figures are 2.323 and 1.492 respectively (Johannsen, 1939). The amorphous character of the obsidian is due to rapid cooling from extrusion temperatures of 600 to 800°C (Friedman et al., 1963). The quick cooling does not allow time for any atoms or ions to group into a regular crystalline arrangement. Rhyolitic obsidian is higher in silica content than other types, and therefore the flow is more viscous, creating thick bodies that weather gradually to form cliffs. Rapid release of dissolved gases during extrusion is responsible for the physical nature of the resultant flow. Most rhyolitic flows grade upward from non-vesicular to vesicular glass, to a surface covering of pumice. Water that is evolved from the flow is degassed in the process of eruption so that only 0.10 - 0.30% by weight remains in the glass. This concentration of juvenile water is a function of water vapor pressure and temperature of the flow (Friedman et al., 1963).

Rhyolitic obsidian is a complex aluminosilicate glass containing small amounts of other elements, usually less than 4% Na and K and less than 3% Fe and Ca, as well as numerous

other minor and trace elements of variable concentrations (Ambrose, 1976). Constituents that contribute more than 0.01% by weight are: SiO<sub>2</sub> (72-76%), Al<sub>2</sub>O<sub>3</sub> (10-15%), Na<sub>2</sub>O (3-5%), K<sub>2</sub>O (1-7%), Fe<sub>2</sub>O<sub>3</sub> (0.5-2.6%), FeO (1-1.8%), TiO<sub>2</sub> (0-0.5%), CaO (0-1.2%), MnO (0-0.1%), MgO (0-0.4%), H<sub>2</sub>O (0.1-0.9%), P<sub>2</sub>O<sub>5</sub> (0-0.1%) (Michels and Bebrich, 1971).

Due to the particular nature of volcanic glass, there are very few pre-Tertiary obsidian sites around. Through time the glass undergoes devitrification by the hydration process, ultimately altering the obsidian to perlite (Marshall, 1961; Friedman et al., 1966). The hydration process continues with increasing internal strain, and the layer spalls off. This occurrence is contingent upon a variety of factors and naturally limits the dating range of the obsidian, although the limits would be more of geological interest than archaeological (Gordus et al., 1967).

The hydration of obsidian is a result of the absorption of water from the ground and surface environment and not from the magmatic process at the time of eruption (Ross and Smith, 1955). The absorption begins as soon as a fresh surface is exposed. The birefringent rim observed in hydrated specimens is the result of this uptake of water without a corresponding change in volume, which causes strain (Ericson et al., 1976). During

burial, the obsidian comes into contact with organic and inorganic contaminants in the soil which produce moisture. When a saturated surface on the glass is formed, there is likely to be leaching and concentration of Na and K along with formations of their hydroxides at the surface. This maintains high surface moisture levels (Ericson et al., 1976). Also, leaching of electrolytes from within the solid glass to the surface causes lowering of vapor pressure at the condensed surface layer and so induces further condensation (Yager and Morgan, 1931). Therefore the hydration rate is probably independent of relative humidity levels.

The actual method of water uptake by glass involves an autocatalytic process. This is because water is a reactant as well as a catalyst. As more water reacts with the glass structure, the diffusion rate is thought to increase (Weyl and Marboe, 1967). Models for the actual method of ion exchange have been suggested (Moulson; Ericson et al., 1976), but as yet are not entirely satisfactory. A mathematical description of a simple ideal diffusion process is the Arrhenius equation. But since the original water and the water of hydration in obsidian are held by chemical means and during hydration the obsidian structure is modified, objections to the use of this equation have been made (Kimberlan, 1976). However, until

diffusion kinetics are better understood, the Arrhenius equation at present appears to be a workable solution.

#### FACTORS AFFECTING HYDRATION RATES

As previously mentioned, the mechanism of leaching and formation of hydroxides allows the hydration process to occur in very wet or very dry climates, with no apparent effect upon the rate by these different atmospheric moisture levels.

It has been suggested that the chemical environment may have a bearing on hydration rate (Pike and Hubbard, 1953; Brüchner, 1971). Diffusions of  $H^+$ ,  $OH^-$ ,  $K^+$  and  $Na^+$  require lower activation energies than the other ions likely to enter the reaction, and most likely reactions with Na and K are the most rapid. Experiments with obsidian showed damage above pH 10, suggesting obsidian is very resistant to chemical attack (Pike and Hubbard, 1953). If obsidian does however suffer chemical attack (which would happen only in the most aggressive environment), etch pits can be seen and the sample can be discarded as unreliable (Ambrose, 1976). Based upon what is known to date, the hydration rate of obsidian is controlled mainly by two factors, the chemical composition of the obsidian and the temperature at which hydration occurs.

Using a method of neutron activation analysis, several researchers were able to determine source sites for Hopewellian obsidian found in Ohio (Griffin et al., 1969; Gordus et al., 1971; Gordus et al., 1967). Elemental composition of artifacts from Hopewell sites in Ohio was determined by this method and compared with compositions of several possible sources in the western United States. A percent ratio of Na/Mn was compared and the Hopewell obsidian was classified into two source areas; one with a Na/Mn ratio of 150, and the other a Na/Mn ratio of 90. In the 90 group, iron concentrations were slightly higher, as were lanthanum and scandium (both present in minute amounts <0,004% by weight). Generally, it appears that Na, K and hydroxyl have a positive effect on hydration and iron and calcium have a negative effect (Ambrose, 1976). Therefore, the 90 group could conceivably hydrate at a slightly slower rate than the 150 group. According to Friedman and Long (1976) SiO<sub>2</sub> content increases the hydration, and CaO and MgO reduce it. In their experiments, Al<sub>2</sub>O<sub>3</sub>, FeO, Na<sub>2</sub>O, and K<sub>2</sub>O had little effect. Griffin et al. (1969) showed that the source of the 150 group was Obsidian Cliff in Yellowstone Park. The source of the 90 group has yet to be determined, although it is also believed to be in Yellowstone Park. Furthermore it is believed that the composition of obsidian in a single flow is relatively

uniform due to its formation by rapid cooling (Gordus et al., 1971).

Of the obsidian tested by Griffin from Mound 25 of the Seip Mound group in Ohio, seven flakes were found to be from the 150 group and one from the 90 group. The obsidian flakes that were checked for hydration rates in this study were also from Mound 25. Therefore, the obsidian could have come from either source, although it seems reasonable to assume it is probably from Obsidian Cliff.

Previously hydration rates were dependent upon correlation with known radiocarbon dates. By making use of the Arrhenius equation and the knowledge that obsidian is source specific, Friedman and Long (1976) were able to liberate hydration rim dating from its radiocarbon dependency. They believe that hydration follows the rate equation:

$$T = (kt)^{1/2}$$

where T is the thickness of the hydration layer, t is the time, and k is a constant. In order to find the constant k, the Arrhenius equation is used:

$$k = Ae^{-E/RT}$$

where  $k$  is the hydration rate ( $\mu^2/10^3$  yrs),  $A$  is a constant,  $E$  is the activation energy (calories per mole),  $R$  is the gas constant (calories per degree per mole), and  $T$  is the absolute temperature (degrees Kelvin). In order to find  $k$ , Friedman and Long (1976) artificially hydrated obsidian from twelve different volcanic extrusions under controlled conditions. The obsidian was hydrated at elevated temperatures and water pressures for various lengths of time, after which a section was cut and the hydration rim thickness measured. The results were plotted on a graph (depth in  $\mu$ , squared versus time in years) (Figure 1). Each slope from each graph is a value for the hydration rate ( $k$ ) of the obsidian at that particular temperature. The natural logs of these values are then plotted versus the inverse of the temperature in Kelvin times 1000 (Figure 2). The slope of this graph, then divided by the gas constant yields the activation energy of the hydration process. The y-intercept of the graph yields  $\ln A$ . The activation energy is different for each different chemical composition. Once the values for  $A$  and  $E$  have been solved for a particular source, the correct temperature must be used to re-solve the equation for  $k$ .

Because of the exponential relation between temperature and hydration rate, a simple average of temperatures cannot



be employed. Instead, the Arrhenius equation must be solved using temperatures to which the obsidian must have been exposed over short time periods, and then the values for  $k$  averaged to obtain one temperature to use in the equation to obtain an average hydration rate over a long time period. This technique for measuring hydration temperature was tried by Friedman and Long (1976), by making periodic temperature measurements on a buried sensor. O'Brien (1971) tried the Pallman technique, an innovative method making use of a temperature dependent sucrose reaction. Ambrose (1976) used a sphere filled with a molecular sieve immersed in water buried at the site for a period of time. The rate of diffusion of water is temperature dependent. A less complicated, though less accurate way to determine average site temperature is to estimate it from records of mean air and soil temperatures made near the site in question. The United States Department of Commerce ususally has these records. The estimated ground temperature of depths from 2 to 10 m in Ohio is  $9.5^{\circ}\text{C}$  (Friedman and Long, 1976). By using this method one assumes that the climate has remained relatively stable since burial of the artifacts. This assumption may be particularly valid in Ohio, over a span of about 2000 years, since it is known the Hopewell grew various temperate crops, particularly maize,

similar to those grown in the region today (Morgan, 1952). Also by the ceremonial use of obsidian by the Hopewell, it is known that rapid burial ensued after a fresh surface had been made, thereby assuring only minor temperature fluctuations.

#### PROCEDURE

Obsidian flakes from Mound 25, Seip Mound Group were obtained from the Ohio Historical Society through the auspices of Ms. Martha P. Otto. The flakes were found during excavation of Hopewell site Mound 25 in Ross County, Ohio (Shetrone and Greenman, 1931). They were buried within the mound at depths of about 5-10 m. Radiocarbon dates from a house pattern near the site indicate occupation around  $230 \pm 80$  AD and  $280 \pm 55$  AD. These dates were determined in 1971, after the radiocarbon dating correction curves had been made known (Suess, 1970).

The flakes were cut perpendicular to the chipped surface by a Felker water trim diamond saw. They were then ground smooth on a Buehler Ltd. 18 inch steel rotating lap with corundum grits grading from 220 to 400 to 600. They were then cemented with epoxy to a standard petrographic slide, and ground with an Ingram thin section cut-off saw and

grinder to approximately 0.003 inch thickness.

With the first thin sections made, trouble with chipping of the edges was noticed. This problem was resolved by first baking the flakes in epoxy to shield the edges from breakage, as suggested by Katsui (1965). If the cut is not made exactly perpendicular to the chipped surface, the hydration rim becomes skewed and may appear thicker than actuality. For that reason only the thinnest bands observed were measured and then averaged when a sample with variable width of hydration was observed.

The slides were observed under a Leitz Ortholux polarizing microscope (500x) and photographed with a 35 mm Leica DBP camera attachment, first with black and white and then with 160 Ektachrome ET 135-36 film. On the same roll of film a Leitz stage micrometer was photographed to serve as a standard of measurement for all photographs of hydration bands on that roll of film. The slide of the stage micrometer was projected onto a piece of plain white paper and single micron intervals were measured and drawn on the paper. Then the slides of the hydration bands were projected over the paper and the width of the rim measured from eight samples. Measurements taken from the samples were averaged to obtain one figure per sample. The variability between the samples was less than 0.10  $\mu$ , and the results were averaged to obtain one figure for the group.

## RESULTS

The data found by Friedman and Long (1976) for Obsidian Cliff obsidian was substituted in the Arrhenius equation:

$$k = Ae^{-E/RT}$$

where  $A = 6.457 \times 10^{15}$ ,  $E = 19,580$  cal/mole,  $R = 1.987$  cal/mole/°K, and  $T = 9.5^\circ\text{C} = 282.5^\circ\text{K}$ ,  $k = 4.6 \mu^2/10^3$  years.  $k$  was then substituted into the equation:

$$T^2 = kt$$

The average thickness ( $T$ ) of the samples examined was  $2.84 \mu$ , thereby solving for  $t = 1753$  years, or  $227 \pm 62$  AD.

## DISCUSSION

The results obtained by this technique fall well within the range of the radiocarbon dates obtained for the site. According to Findlow and De Atley (1976), the method of photographic enlargement by slide projection used here reduces the error in measurement of the bands to  $0.05 \mu$ . The  $0.05 \mu$

error factor includes shrinkage of the film, variations in the width of the marker used to draw the scale, variations in the accuracy of the ruler used to measure the bands and error in the accuracy of the stage micrometer. Incorrect slide preparation is also an error factor, although photographing the slide enables the observer to study it in great detail for any possible signs of chipping of the rim or other problems caused by preparation. Chipped areas can be avoided during measurement. There appears to be, however, a problem associated with the use of epoxy rather than Canada balsam for the cementing procedure. The epoxy created the appearance of two hydration rims, although the true rim, seen on the outer edge of the sample (Plate I), can be determined with careful scrutiny.

Several years prior to this study of the obsidian from Seip Mound, radiocarbon dates had been made from wood samples from an adjacent site. Therefore the probable date at the time of the chipping of the obsidian is known, and a rough idea of how wide the hydration band should be could be estimated. Testing of obsidian from a site not previously radiocarbon dated should be a project for further study.

Generally, the Midwest is an ideal area for applying the obsidian hydration dating technique. The use of obsidian

by prehistoric populations in this region for ceremonial purposes generally meant immediate burial of the artifact after chippage. The flakes were usually incorporated into the fill dirt of the constructions. The climate has apparently not changed markedly over the last few millennia and the average site temperature can easily be obtained through extrapolation of modern records. A remaining complication is that the elemental composition of the obsidian must be determined, and the hydration rate for that glass found prior to any attempts of dating.

Obsidian hydration rim dating is a quick, inexpensive means of determining the age of prehistoric cultures. As research continues, and the error factors are reduced, this method of age determination could become indispensable to the archaeologist.

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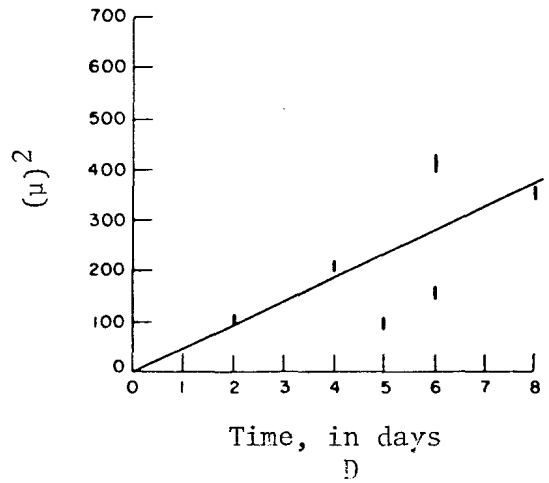
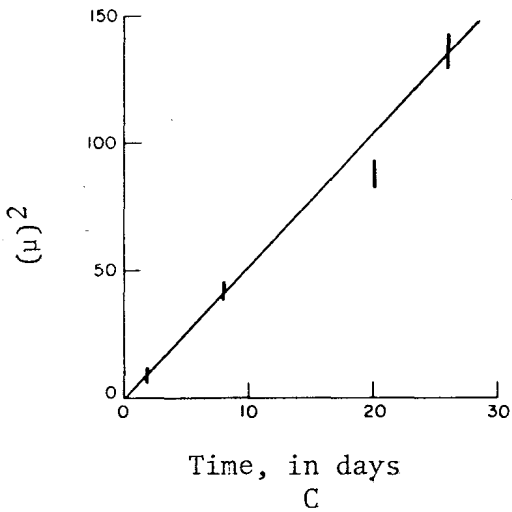
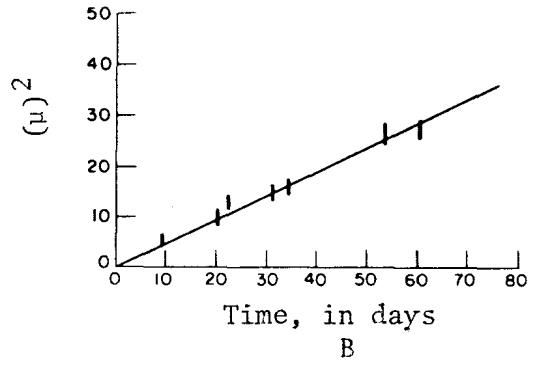
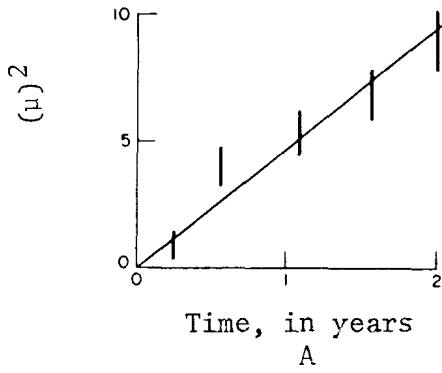


Figure 1. Experimental hydration curves for obsidian from Kerlingarfjoll, Iceland. Hydration conditions were: (A) 95°C and 1 kg/cm<sup>2</sup> H<sub>2</sub>O pressure; (B) 150°C and 3.5 kg/cm<sup>2</sup>; (C) 195°C and 14 kg/cm<sup>2</sup>; and (D) 245°C and 350 kg/cm<sup>2</sup>. The slopes of the graphs are values of the hydration rate (k). The same method was used to determine k values for obsidian from Obsidian Cliff, Yellowstone Park. From Friedman and Long (1976).

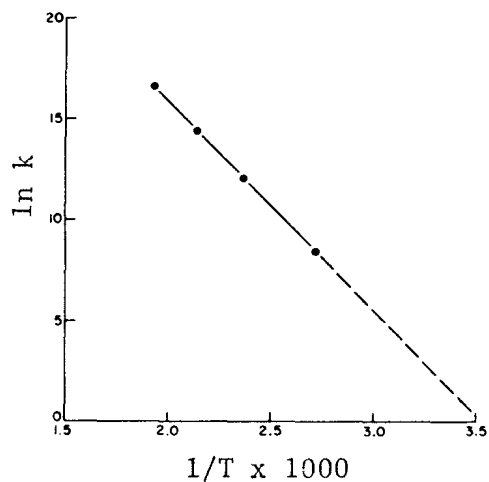


Figure 2. Natural logs of  $k$  values obtained for obsidian from Kerlingerfjoll, Iceland, plotted against inverse of the temperature in degrees Kelvin. The slope of this graph divided by the gas constant (1.987 cal/mole/°K) gives the activation energy ( $E$ ). The y-intercept yields  $\ln A$ . The same method was used to obtain  $A = 6.457 \times 10^{15}$  and  $E = 19,580$  cal/mole for obsidian from Obsidian Cliff, Yellowstone Park. From Friedman and Long (1976).

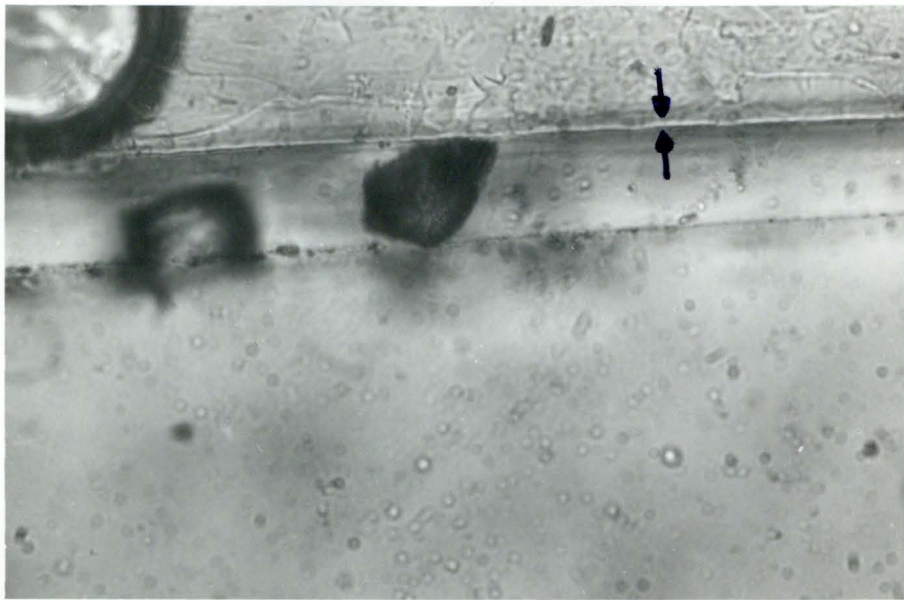
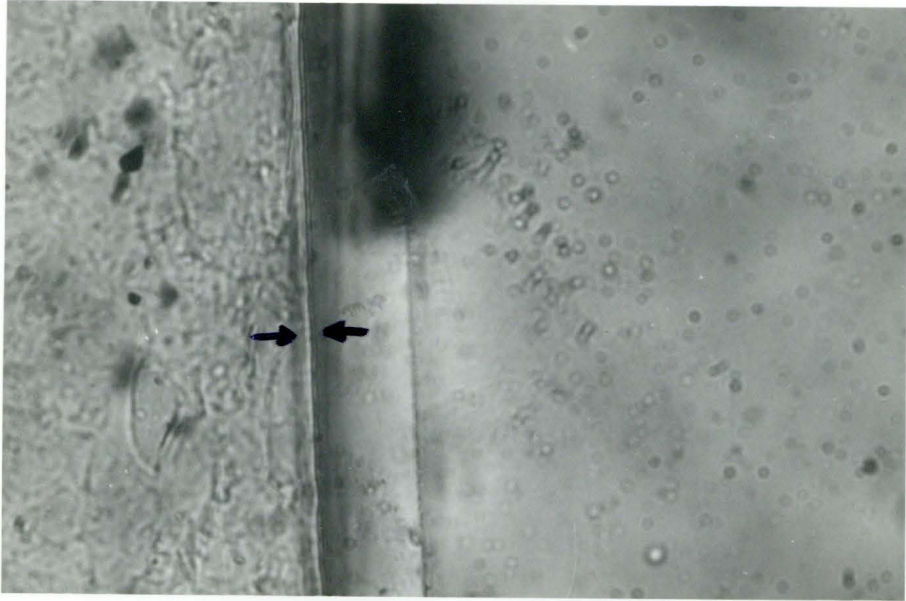


Plate I. Photomicrographs of obsidian hydration bands (arrows) from two different samples from Seip Mound, Ohio. Larger band is epoxy.